SUPERPLASTIC BEHAVIOR OF A Fe-Mn-Al AUSTENITIC STAINLESS STEEL

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Abstract. The superplastic behaviour of Fe-Ni-Cr duplex stainless steel system has been the subject of several investigations in the last two decades, with superplastic forming applications already existing in practice. A rare example of study of superplasticity on a Fe-Mn-Al steel was presented by Toscano (1983) showing some possibility of exploring the potential use of such materials in this regime for temperatures higher than 700°C. The present work was programmed to explore the occurrence of this behaviour in a similar steel with systematic hot tensile tests carried out in the range from 600 to 900°C and strain-rates varying from 8 x 10^{-5} to 8 x 10^{-2} s^{-1}. The variation in sensitivity of stress with strain rate ($\sigma = C\,\dot{\varepsilon}^{m}$) was observed using distinct specimens pulled until rupture under different combinations of crosshead speed and temperature, as well as single specimens subjected to a sequence of crosshead speed changes during the hot tensile test. At 800 and 900°C the maximum values of **m** ($m = dLog\sigma/dLog\dot{\varepsilon}$) were found to be about 0.57 and 0.66 respectively, which confirms the material susceptibility to superplastic behaviour. The maximum elongation values were observed to stay around 300% obtained with the lowest strain rate level of 8 x 10^{-5} s^{-1} at 850°C.

Keywords: *Fe-Mn-Al steel, hot tensile test, strain-rate sensitivity, superplasticity.*

1. Introduction

It is well known that superplastic materials exhibit usually a three-stage relationship in the steady-state strain rate ($\dot{\varepsilon}$) dependence of the applied stress (σ) [1]. These three ranges are named regions I, II and III, respectively. The most important superplastic characteristics associated to high elongations occur in region II, with progressive drop in superplastic characteristics in both regions I and III, as shown schematically in Figs. 1a and 1b, according to Langdon (1982). There are two kinds of mechanical tests to explore the superplastic properties of metals: a) tensile tests on constant crosshead speed (or constant strain rate) machines where the flow stresses are measured as function of the strain rates and are related by the expression: $\sigma = C\,\dot{\varepsilon}^{m}$, where C is a constant including the temperature dependence, and m is the strain rate sensitivity exponent ($m = dLog\sigma/dLog\dot{\varepsilon}$); b) tensile tests on creep machines at constant load (or constant stress), where the strain rates are measured as function of the imposed stresses, being related by the expression: $\dot{\varepsilon} = A\,\sigma^{n}$, with $n = 1/m$. Values of m ranging from 0.35 to 0.8 are generally considered to produce superplastic behaviour.

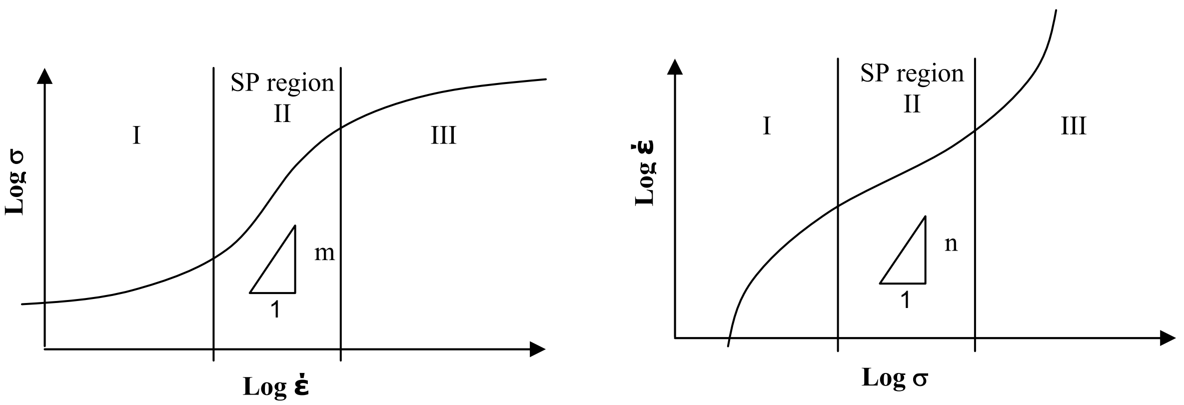

Figure 1 – Schematic illustration of the two different procedures used to plot the mechanical data of superplastic materials: **a)** stress vs strain rate and **b)** strain rate vs stress.

The Fe-Mn-Al stainless steels may exhibit good combination of properties like mechanical strength, ductility, corrosion/oxidation resistance and lower density, being considered as alternative materials to the Fe-Ni-Cr stainless steels in some applications. Several studies have been reported since the 1960s involving the characterization of their conventional properties at room temperature. The mechanical behaviour of these alloys at high temperatures, however, remains largely unexplored, with very little information existing in the literature on creep and superplastic properties, for instance. On the other hand the superplastic behaviour of Fe-Ni-Cr duplex stainless steel system has been the subject of several investigations in the last two decades, with superplastic forming applications already existing in the aerospace industry. To our knowledge the only study of superplasticity on a Fe-Mn-Al austenitic steel was presented by Toscano (1983) showing some possibility of exploring the potential use of such materials in this regime for temperatures higher than 700°C. The steel was produced with a fully austenitic structure being submitted to high reduction in thickness by cold rolling. Isothermal tensile tests carried out at a fixed crosshead speed Vc = 0.5 mm/min have shown a drop in the yield and tensile strength of the material from 800 to 1000K , followed by an increase in hardness in the same temperature range, due to the precipitation of the β-manganese phase, as shown in Fig.2. During the hot tensile tests, from 793K, incipient precipitation of the β-manganese phase occurs in the austenite grain boundaries, which promotes a very a fine grain size structure from 913K. The appearance of superplastic behaviour in Fe-Mn-Al steel is explained by the movement of the rigid particles of β-phase in the "non-Newtonian fluid" of the matrix. After this work, apparently, there are no other investigations reported in literature concerning superplastic behaviour of Fe-Mn-Al stainless steels.

The present work was programmed to explore the occurrence of this behaviour in this type of steel with systematic hot tensile tests carried out on a constant crosshead speed machine in the range from 600 to 900°C involving initial strain-rates from 8.3×10^{-5} to 8.3×10^{-2} s^{-1}.

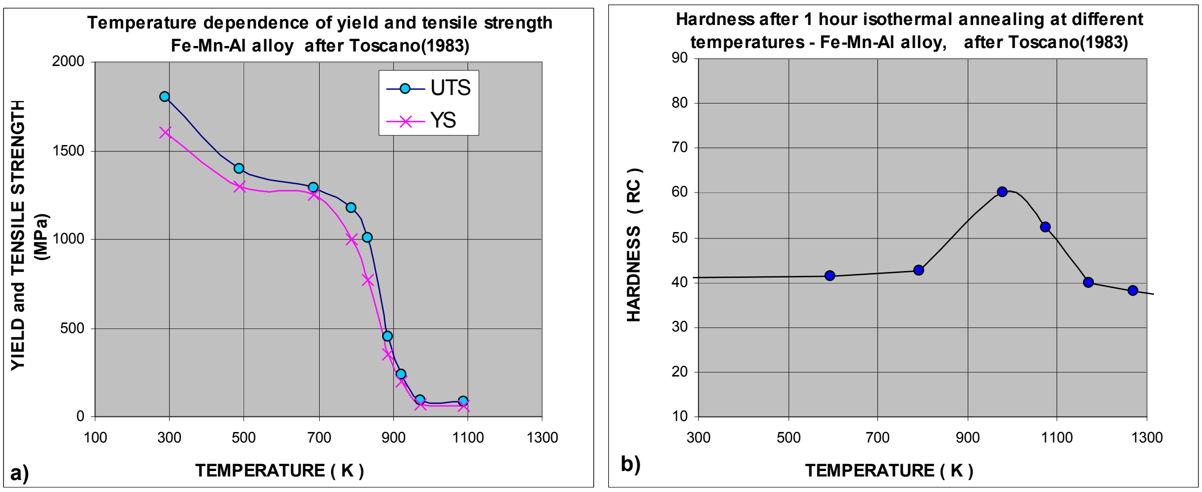

Figure 2 – Fe-Mn-Al steel data: **a)** Temperature dependence of Yield and Tensile Strength at different temperatures; **b)** Variation in Hardness after 1 hour isothermal annealing at various temperatures followed by cooling under vacuum. Adapted from Toscano (1983).

2. Experimental Procedures

The material was prepared in the form of ingots weighing about 3.5 kg with approximately 50 x 50 x 220 mm each. The chemical composition was determined as: Fe– 24.5Mn– 6.5Al– 1.5Si– 1.1C– 0.009P– 0.016S (wt%). The ingot was first submitted to solution heat-treatment at 1050°C for 24 hours, followed by quenching in oil. Grinding operation was used to square all the faces before sectioning the sample in two slabs with about 25 x 50 x 200 mm each. Hardness of the material at this condition was 286 HV_{30}. The slabs were subjected to three series of cold rolling steps of low deformation followed by heat treatments of 1050°C during 1 hour. The accumulated deformation levels after each cold rolling stage corresponded to about 25, 50 and 75% reduction in thickness. After the last solution treatment the sample was cold rolled continuously until its final shape of a stripe with 1mm final thickness. Figure 3 illustrates the sequence of shape changes at each stage in the preparation of the material.

Tensile samples were machined from the stripes in the rolling direction, as shown in Figure 4a, having a nominal gauge length Lo = 10 mm and gauge width w = 3.0 mm, similar to the samples used by Toscano (1983). Tensile tests were carried mainly at 600°C, 700°C, 800°C and 900°C, with four crosshead speed levels, namely: Vc = 0.05, 0.5, 5 and 50 mm/min, corresponding to initial strain rates of : 8.3×10^{-5}, 8.3×10^{-4} , 8.3×10^{-3} and 8.3×10^{-2} s^{-1}, respectively. The hot tensile tests were carried out on a universal Instron machine model 5500R with a tubular electric resistance furnace.

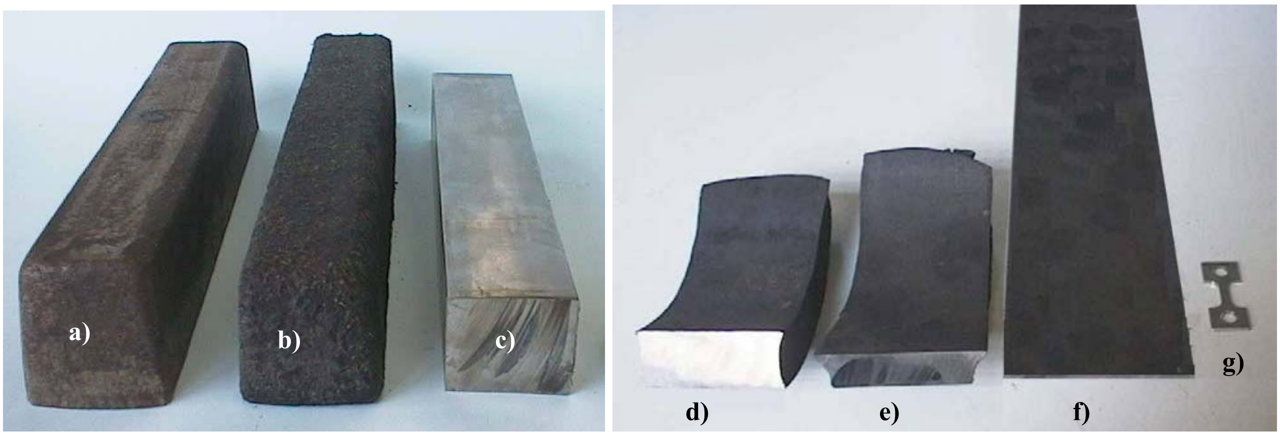

Figure 3 – a) Ingots of Fe-Mn-Al steel as cast; **b)** Ingot after solution treatment at 1050°C, during 24 h, quenched in oil; **c)** After grinding operation prior to rolling; **d)** and **e)** slab after different rolling stages; **f)** final shape as stripe with about 1mm thickness; **g)** tensile specimen machined in the rolling direction.

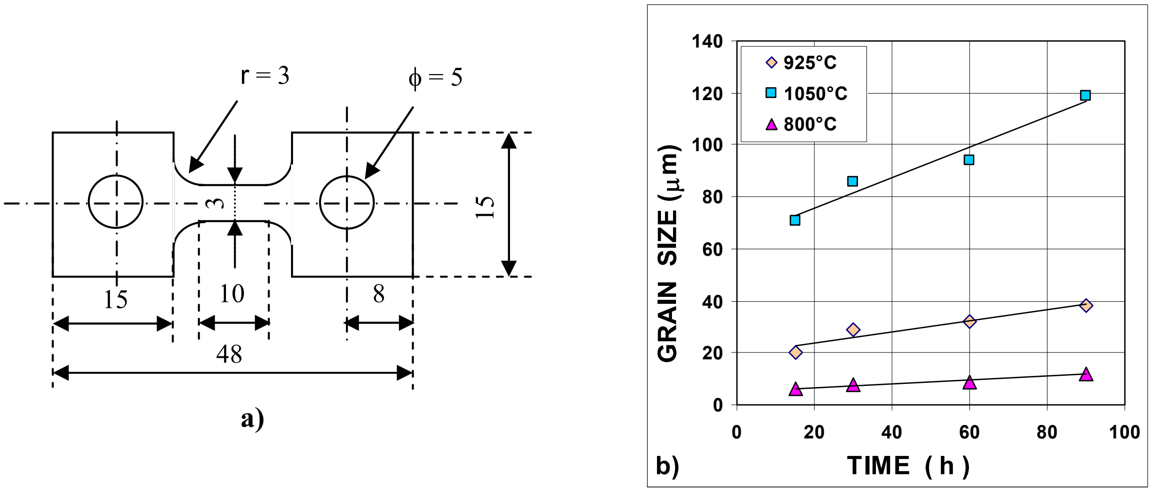

Figure 4 – **a)** Specimen for the hot tensile tests; **b)** Variation of grain size with temperature and time during annealing for Fe-Mn-Al steel sheet with 0.5 mm thickness, according to Cintho (1988).

The temperature stability during all tests was about $\pm$1ºC, maintained by P.I.D controllers. There was an interest in carrying out some extra tests: a) one at room temperature (25ºC) at Vc = 0.5mm/min (the same crosshead speed used by Toscano (1983) in his tests); b) four others at 750, 825, 850 and 875°C at Vc = 0.05mm/min.

The variation in sensitivity of stress with strain rate was observed using *distinct specimens* for each combination of crosshead speed with temperature, as well as *single specimens* subjected to several crosshead speed changes during a certain temperature level.

3. Results and Discussion

Figure 5a show examples of typical curves of the hot tensile tests at the same crosshead speeds and different temperatures, and Fig. 5b curves at the same temperature and different crosshead speeds. At room temperature the material shows very high yield and tensile strengths in the cold rolled condition, as reported by Toscano (1983). As the temperature increases the strength of the material becomes more and more strain rate sensitive.

Figure 6a presents the variation of the tensile strength with temperature at the different crosshead speeds. Figure 6b shows a comparison between results of this work with those by Toscano (1983) obtained at the crosshead speed Vc = 0.5mm/min, indicating good agreement of the data.

Figures 7a, 7b, 7c and 7d show the sensitivity of true stress with true strain rate at the nominal stress peaks, using different specimens for each crosshead speed at 600°C, 700°C, 800°and 900°C, respectively. Although only four different values of strain rate were explored in each case, the data shows approximately the typical sigmoidal trend mentioned in literature for superplastic materials. The maximum value of m was estimated in each case. At 900°C the data were clearly not sufficient to show any sign of region I.

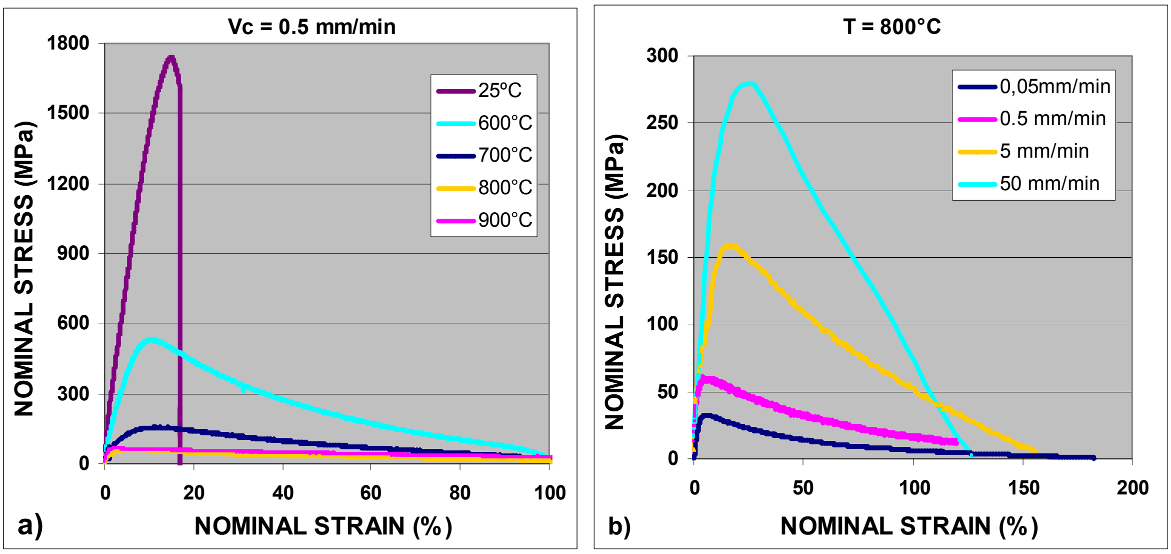

Figure 5 – Typical Stress versus Strain curves for the Fe-Mn-Al steel used in this work: **a)** at the same crosshead speed and different temperatures; **b)** at the same temperature and different crosshead speeds.

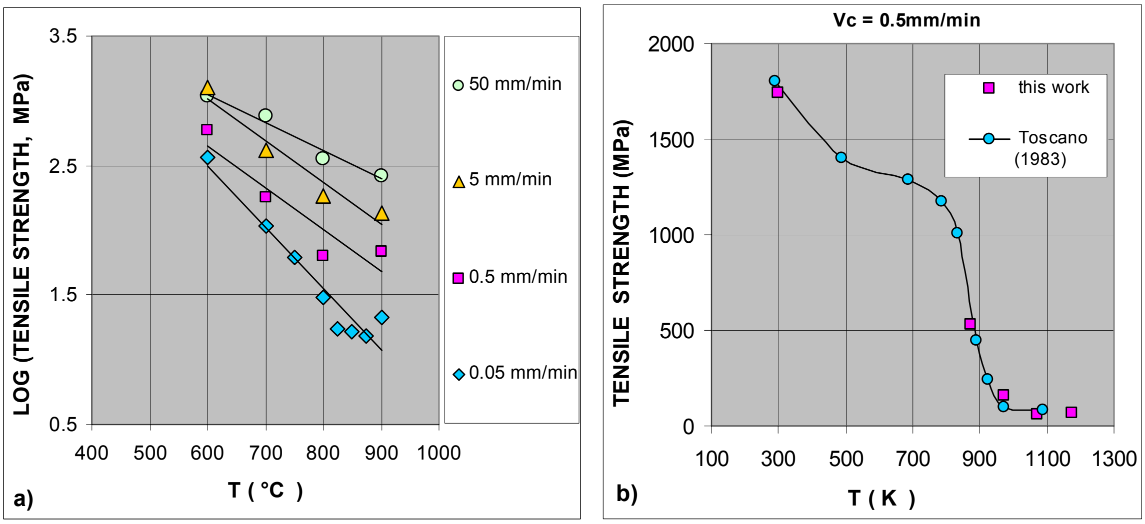

Figure 6 – **a)** Variation of Tensile Strength with Temperature for different crosshead speeds; **b)** Comparison between results of this work with data by Toscano (1983) at the crosshead speed Vc = 0.5mm/min.

Figure 8a shows a comparison between the sensitivity of stress with strain rate at the four temperature levels using distinct specimens.

Figure 8b presents a typical nominal stress versus nominal strain curve obtained with a single specimen submitted to changes in crosshead speed after reaching the nominal peak stresses. Figures 9a, 9b, 9c and 9d show the sensitivity of true stress with true strain rate at 600°C, 700°C, 800°C and 900°C, respectively, using single specimens in each case. The curves also show the sigmoidal trend similar to the Figs. 7a, 7b, 7c and 7d. The maximum value of **m** could better estimated due to the higher number of points obtained in these kind of tests. At 700°C it was not possible to make the stress changes at the lower crosshead speeds and the data are certainly not well situated in region II so that the **m** value mentioned in the figure is lower the real maximum value.

The maximum strain rate sensitivity exponent is observed to increase from about 0.28 to 0.66 as temperature increases from 600 to 900°C, respectively. Figure 10a and 10b show a comparison between the sensitivity of true stress with true strain rate, using single specimens and distinct specimens at 800 and 900°C, respectively, demonstrating good agreement between the results from the two kinds of testing techniques.

Figures 11a and 11b show the variation of the strain rate sensitivity exponent with strain rate using single specimens at 800 and 900°C, with the estimated maximum **m** values of 0.57 and 0.66, respectively. The probable regions of superplastic regime are schematically indicated in each case, assuming a certain pattern of symmetry for the

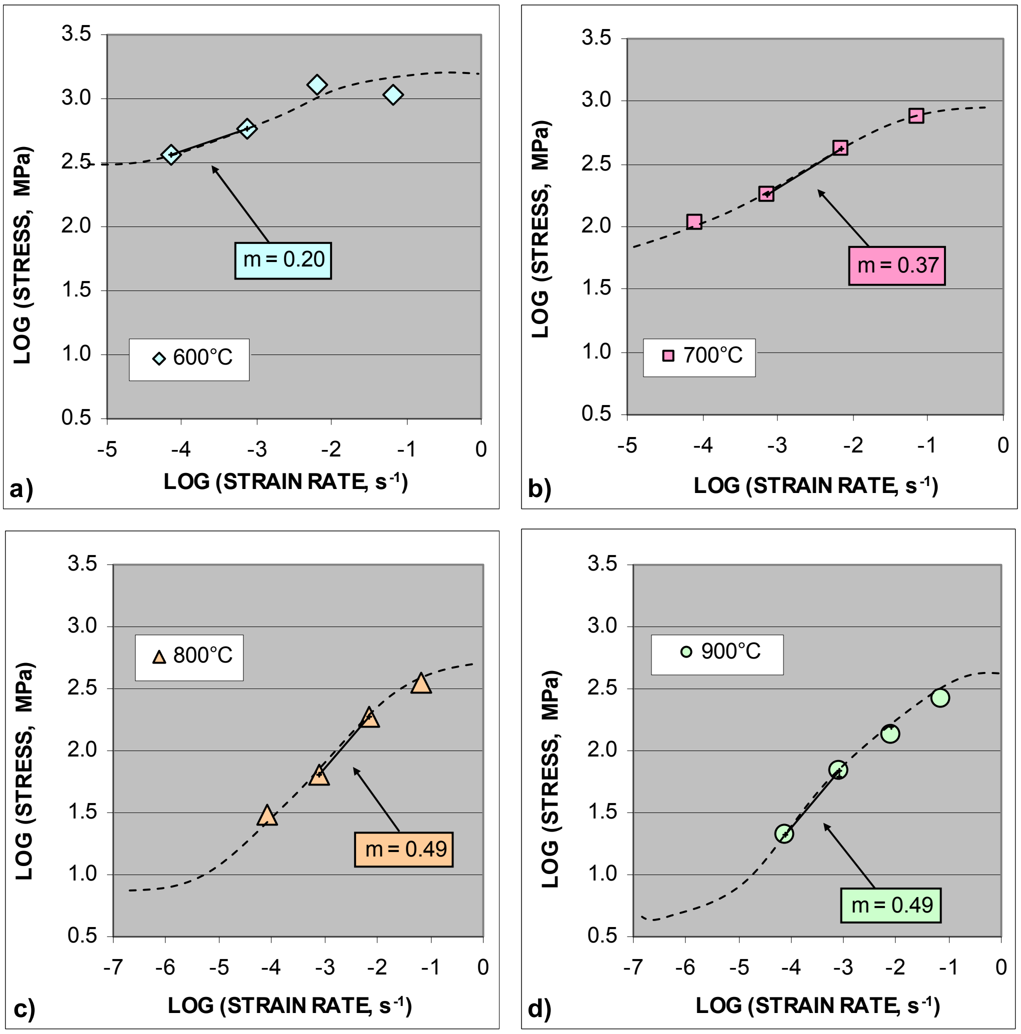

Figure 7 – Sensitivity of true stress with true strain rate at the nominal stress peaks, using different specimens for each crosshead speed at : **a)** 600°C; **b)** 700°C ; **c)** 800°C ; **d)** 900°C.

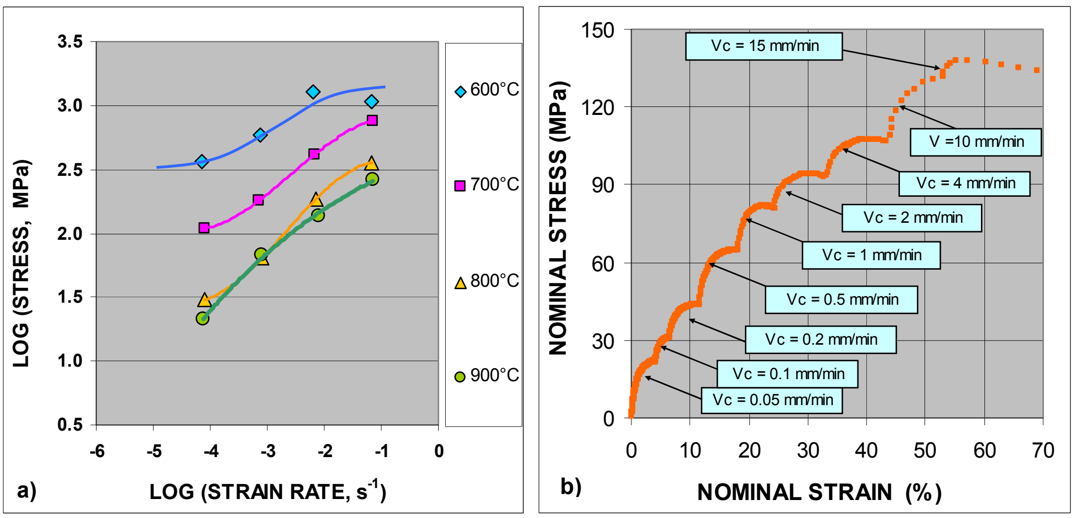

Figure 8 – **a)** Comparison between the sensitivity stress with strain rate using distinct specimens at different temperatures and crosshead speeds **; b)** Typical nominal Stress versus Strain curve with changes in crosshead speed after reaching the peak stress. T = 800°C.

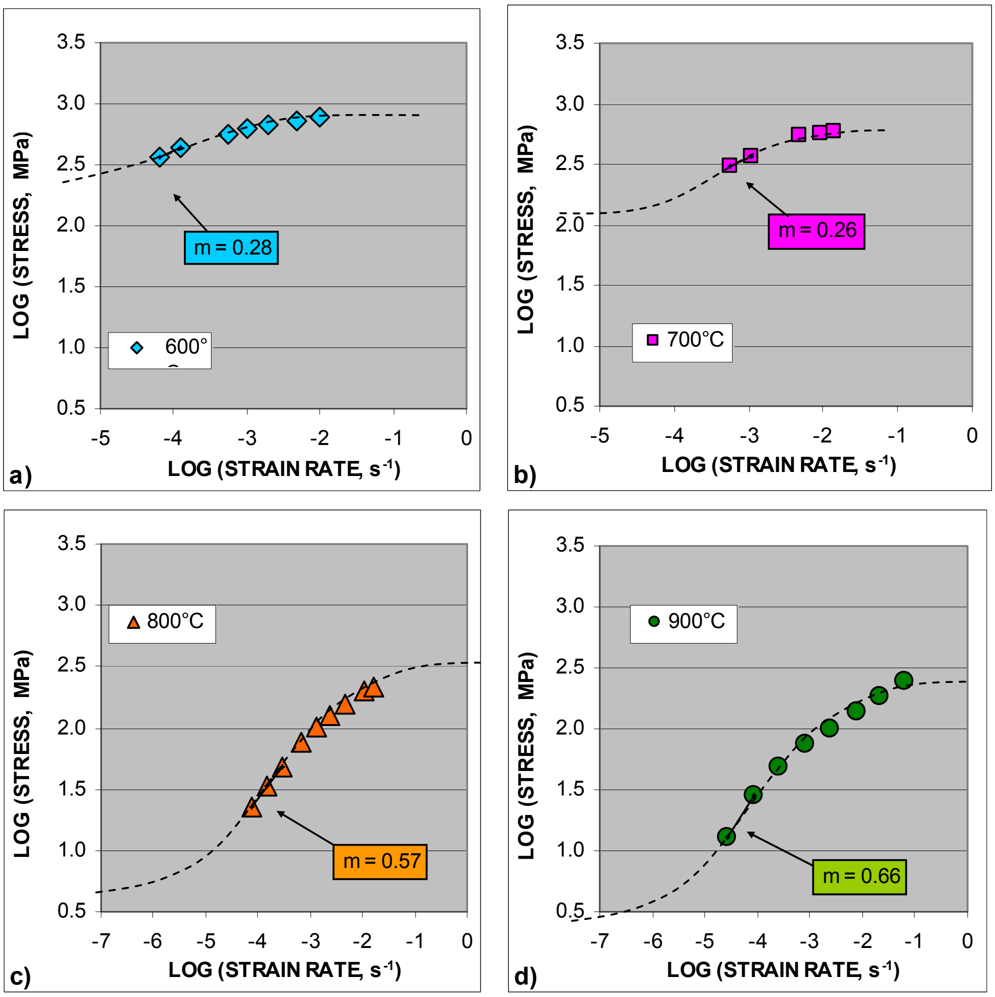

Figure 9– Sensitivity of true stress with true strain rate at the nominal stress peaks, using a single specimen subjected to changes in crosshead speed at : **a)** 600°C ; **b)** 700°C; **c)** 800°C; d) 900°C.

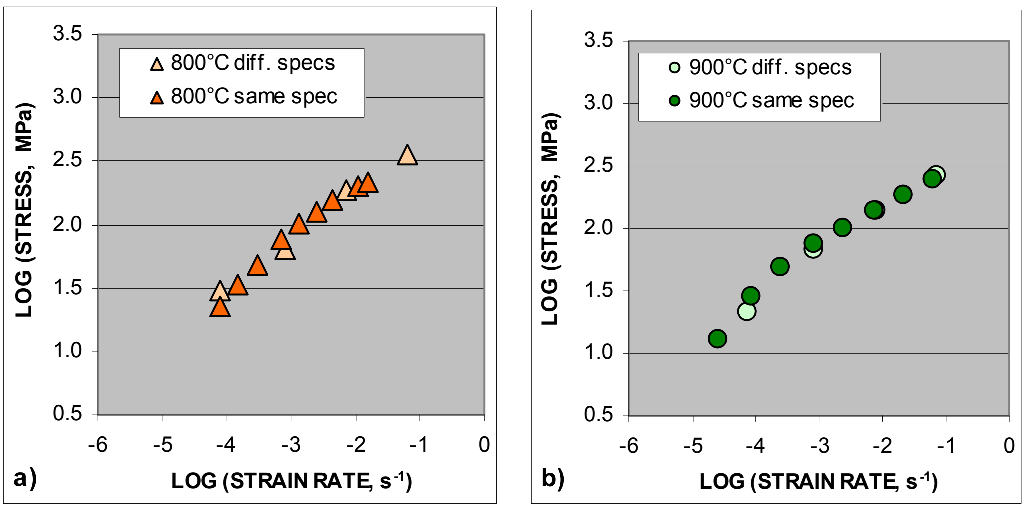

Figure 10 – Comparison between the sensitivity of stress with strain rate, using single specimens and different specimens at: **a)** 800°C; **b)** 900°C.

data, as usually reported by various authors (Langdon, 1982, Maehara, 1985, Bae and Ghosh, 2000).

Figure 12a shows a comparison between the variation of the strain rate sensitivity exponent with strain rate at 800 and 900°C, respectively. The curve at 900°C exhibits a maximum **m**-value higher than the curve at 800°C, as expected. However, the curve at 900°C is clearly situated at the left hand side of the 800°C curve, contrary to the normal trend expected for the effect of temperature on such data (Bae and Ghosh, 2000). Normally, the increase in grain size displaces the curve to the left and lower its maximum **m**-value. More tests and studies involving microstructural observation on the superplastic deformed specimens would be necessary to confirm and understand this effect.

Figure 12b presents the trend for increase in the strain rate sensitivity exponent with temperature. The figure indicates that the maximum **m**-value at 700°C is expected to be higher than the value measured as commented previously.

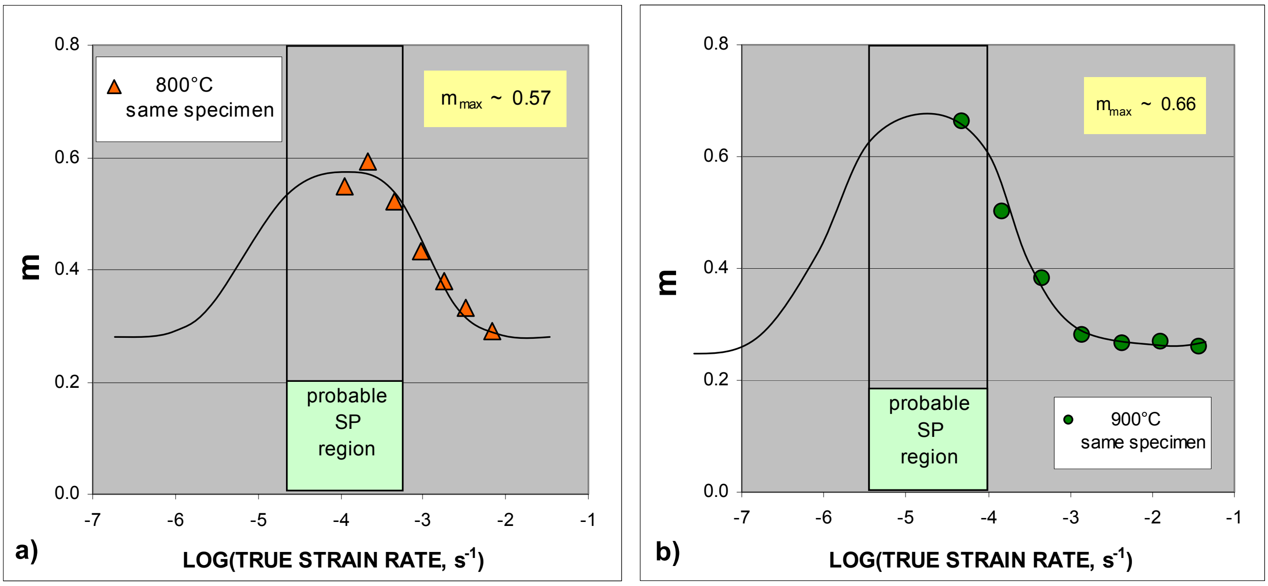

Figure 11 – Variation of the strain rate sensitivity exponent with strain rate using single specimens at: **a)** 800°C; **b)** 900°C. Solid lines are schematic representation of possible trend of the data.

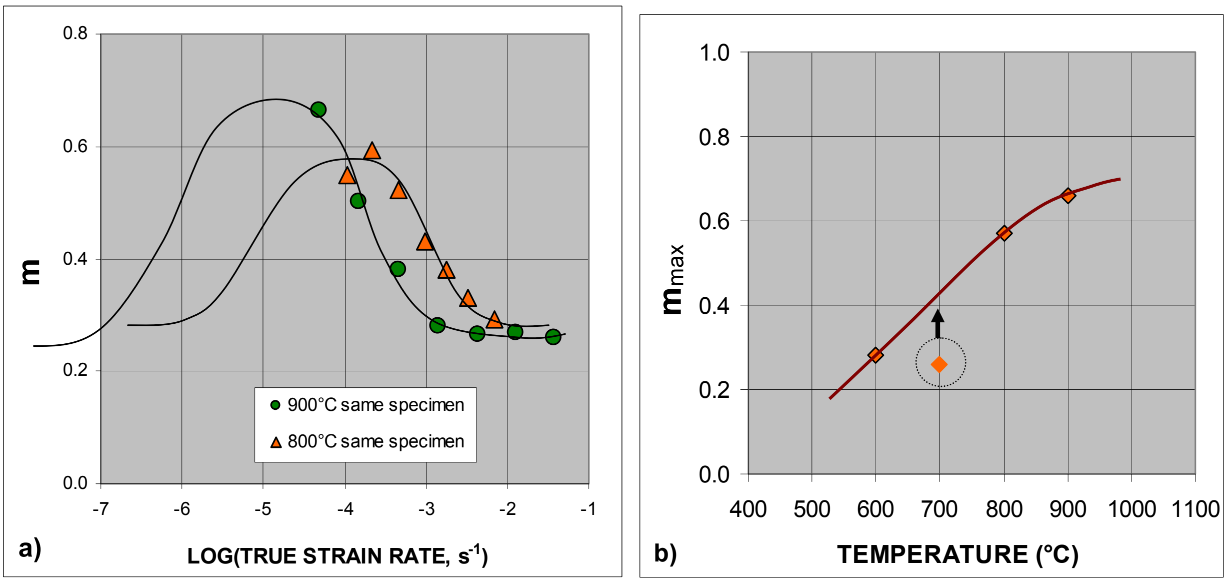

Figure 12 – a) Comparison between the variation of the strain rate sensitivity exponent with strain rate at 800°C and 900°C; **b)** Variation of the maximum strain rate sensitivity exponent with temperature.

Figure 13 illustrates the appearance of some tensile specimens with different elongations after rupture at various temperatures and the crosshead speed Vc = 0.05 mm/min, where the superplastic effect was more prominent.

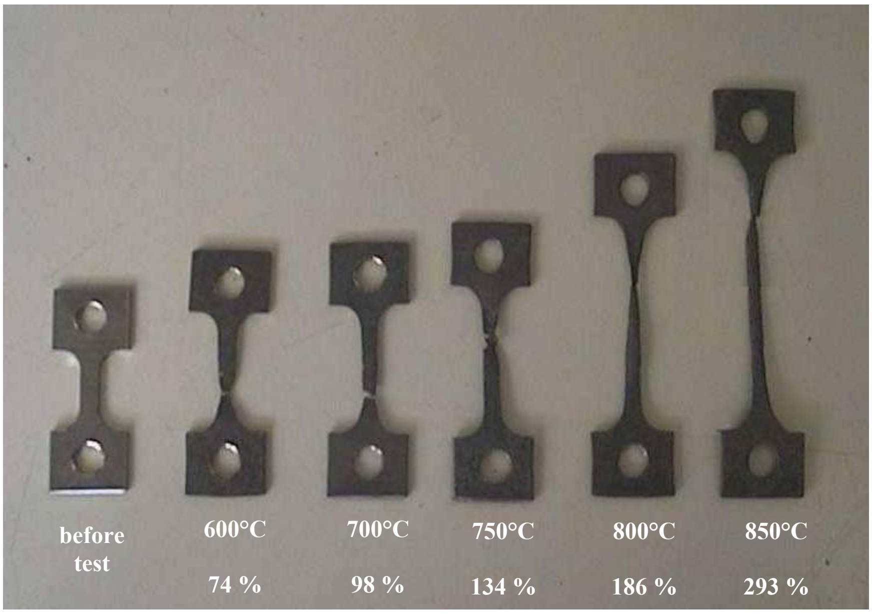

Figure 13 – Tensile specimens having different elongation until rupture at different temperatures and same crosshead speed Vc = 0.05 mm/min.

4. Conclusions

The highest elongation value obtained in the present work was about 300% for the specimen tested at 850°C at $8x10^{-5}$ s^{-1}. Although the values of the strain rate sensitivity exponent are relatively high (0.57 to 0.66) in the range from 800 to 900°C indicating possibility of superplastic behaviour, the values of final elongation were a little below the expectation, considering estimated values of about 500% at 800°C at 0.5mm/min from the work of Toscano (1983). Studies on cold rolled commercial duplex stainless steels reveal that elongations higher than 700% may be obtained at temperatures above 950°C (Zhang et al, 2003) or even above 1500% depending on the steel composition and thermomechanical processing (Maehara, 1985, Sagradi et al., 1998).

This work represents a first assessment of the superplastic properties in this material and will be complemented by a new series of of tests to define the better combination of temperature and strain rate conditions where the material could show better superplastic behaviour. The two kinds of tensile tests will be performed in a broader crosshead speed range from 0.005 mm/min to 50mm/min, including higher temperatures levels of 950 and 1000°C.

5. Acknowledgements

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6. References

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